

AD-A032 084

FRANK J SEILER RESEARCH LAB UNITED STATES AIR FORCE --ETC F/G 7/4
THE ELECTROLYTIC REMOVAL OF ALUMINUM FROM A TWO-PHASE ALUMINUM---ETC(U)
NOV 76 J C NARDI, C L HUSSEY, L A KING

UNCLASSIFIED

FJSRL-TR-76-0018

NL

1 OF 1
ADA032084



AD A032084

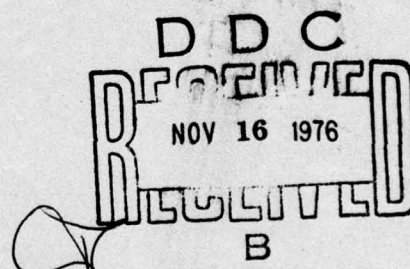


FRANK J. SEILER RESEARCH LABORATORY

FJSRL TECHNICAL REPORT-76-0018
NOVEMBER 1976

THE ELECTROLYTIC REMOVAL OF
ALUMINUM FROM A TWO-PHASE
ALUMINUM-TRIALUMINUM NICKELIDE
MATRIX

PROJECT 2303



APPROVED FOR PUBLIC RELEASE;
DISTRIBUTION UNLIMITED.



AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE

NOTICE

When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Inquires concerning this document should be addressed to the Frank J. Seiler Research Laboratory (FJSRL/NC), U.S. Air Force Academy, Colorado 80840. Phone - 303/472-2655.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151

ACCESSION for	
NTIS	White Section <input checked="" type="checkbox"/>
ORC	Blue Section <input type="checkbox"/>
UNANNOUNCED	<input type="checkbox"/>
JUSTIFICATION	
BY	
DISTRIBUTION/AVAILABILITY NOTES	
Dist.	AVAIL. and/or R.
A	

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER FJSRL-TR-76-0018	2. GOVT ACCESSION NO. ADA	3. PERFORMING ORG. CATALOG NUMBER	
4. TITLE (and Subtitle) The Electrolytic Removal of Aluminum From a Two-Phase Aluminum-Trialuminum Nickelide Matrix,		5. TYPE OF REPORT & PERIOD COVERED Final Technical Report, July 1975 to September 1976	
7. AUTHOR(s) John C. Nardi, Charles L. Hussey, Lowell A. King John K. Erbacher		8. CONTRACT OR GRANT NUMBER(s)	
9. PERFORMING ORGANIZATION NAME AND ADDRESS F. J. Seiler Research Laboratory (FJSRL/NC) Air Force Systems Command U. S. Air Force Academy, Colorado 80840		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS New, 61102F/2303/F2/08 Old, 61102F/7903/02/08	
11. CONTROLLING OFFICE NAME AND ADDRESS F. J. Seiler Research Laboratory (FJSRL/NC) Air Force Systems Command U. S. Air Force Academy, Colorado		12. REPORT DATE November 1976	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 2303, 7903		13. NUMBER OF PAGES 31	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		15. SECURITY CLASS. (of this report) UNCLASSIFIED	
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) F2, 02		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A	
18. SUPPLEMENTARY NOTES			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Aluminum Anodization Trialuminum Nickelide Molten Salts Aluminum Chloride			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The selective electrolytic removal of aluminum from a two-phase matrix of aluminum containing an aligned microstructure of a eutectic alloy, Al ₃ Ni, has been demonstrated feasible in aluminum chloride containing molten salts. Both the 1:1 molar ratio aluminum chloride-sodium chloride melt at 175°C and the 2:1 aluminum chloride-1-ethylpyridinium bromide melt at 23°C were found suitable for this separation. The anodic decomposition potential of Al ₃ Ni in these melts was			

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

319920

JP

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

estimated using linear sweep voltammetry. An apparatus of novel design was used to provide a continuously flowing electrolyte stream past the matrix sample effecting an efficient removal of aluminum.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

SRL-TR-76-0018

THE ELECTROLYTIC REMOVAL OF ALUMINUM FROM A
TWO-PHASE ALUMINUM-TRIALUMINUM NICKELIDE MATRIX

John C. Nardi
Charles L. Hussey
Lowell A. King
John K. Erbacher

TECHNICAL REPORT SRL-TR-76-0018

NOVEMBER 1976

Approved for public release; distribution unlimited

DIRECTOR OF CHEMICAL SCIENCES
FRANK J. SEILER RESEARCH LABORATORY
AIR FORCE SYSTEMS COMMAND
U.S. AIR FORCE ACADEMY, COLORADO 80840

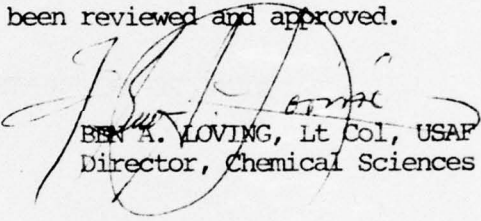
FOREWORD

This report was prepared by the Directorate of Chemical Science, Frank J. Seiler Research Laboratory, United States Air Force Academy, Colorado. The work was initiated under Project No. 2303, "Chemical Synthesis and Characterization," Task No. 2303-F2, "Physical Chemistry and Electrochemistry," Work Unit No. 2303-F2-08, "Aluminum Matrix Separation."

The report covers work conducted from July 1975 to September 1976.

The authors wish to acknowledge the financial assistance rendered by the Rome Air Development Center, Hanscom Air Force Base, Massachusetts.

This technical report has been reviewed and approved.



BEN A. LOVING, Lt Col, USAF
Director, Chemical Sciences

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
Introduction	1
Experimental	3
Results and Discussion	6
Conclusion	10
References	11

FIGURES

<u>FIGURE</u>		<u>PAGE</u>
1	Cyclic Voltammograms of the 2:1 AlCl_3 -EPB and 1:1 AlCl_3 -NaCl Melts	12
2	Three Electrode Cell Used for Cyclic Voltammetry, Linear Sweep Voltammetry, and Al- Al_3Ni Electrolysis Experiments	14
3	Centrifugally Induced Stirring Device	16
4	Anodic Linear Sweep Voltammograms for Al and Al_3Ni Electrolysis in the 2:1 AlCl_3 -EPB Melt at 23°C	18
5	Anodic Linear Sweep Voltammograms for Al and Al_3Ni Electrolysis in the 1:1 AlCl_3 -NaCl Melt at 175°C	20
6	Microphotograph of Al- Al_3Ni Matrix Rod Prior to Electrolysis	22
7	Microphotographs of Al- Al_3Ni Matrix Rod Surface After Anodization at 0.16 A/cm^2 in a Stirred Solution of 2:1 AlCl_3 -EPB at 23°C	24
8	Microphotograph of Al- Al_3Ni Matrix Rod Surface After Anodization at 0.54 A/cm^2 in a Stirred Solution of 2:1 AlCl_3 -EPB at 23°C	26
9	Microphotograph of Al- Al_3Ni Matrix Rod Surface After Anodization at 0.10 A/cm^2 with Centrifugally Induced Stirring in 1:1 AlCl_3 -NaCl at 175°C	28
10	Microphotograph of Al- Al_3Ni Rod Surface After Anodization Illustrating Void Introduced into Rod During Growth of Two Phase Matrix	30

INTRODUCTION

The growth of an aligned microstructure of a controlled eutectic alloy trialuminum nickelide (Al_3Ni) has been investigated by several workers as a means of providing tensile strength enhancement in aluminum (1). A practical method of producing Al_3Ni whiskers in an aluminum matrix utilizes a unidirectional solidification technique (1-3). Small diameter fibers composed of Al_3Ni are also expected to find useful application in industrial and military microelectronics if they can be removed undamaged from the aluminum matrix in which they are imbedded. Investigators of the Rome Air Development Center (RADC), Hanscom AFB, MA, have succeeded in growing Al_3Ni fiber bundles longitudinally in a rod shaped aluminum matrix with a process which shows promise for industrial pilot plant scale-up (4). Thus, the development of a practical means of removing and separating aluminum from a two phase $\text{Al}-\text{Al}_3\text{Ni}$ matrix without damage to the Al_3Ni fibers promises to solve a fundamental problem in their mass production.

Previous attempts using aqueous electrolytes or acid etches for removing or dissolving aluminum from an $\text{Al}-\text{Al}_3\text{Ni}$ matrix have almost always been accompanied by gas evolution resulting in "birdsnesting" of the fiber bundles as they are exposed (4). Another problem reported to be encountered with aqueous acid etches is low selectivity of attack; i.e., the Al_3Ni fibers are dissolved as well as the bulk aluminum (4). An electrolyte that would sustain aluminum electrolysis without gas evolution while permitting selective attack on the bulk aluminum phase would be a potential way to circumvent these problems.

Aluminum has been anodized in aprotic media (5,6), but generally the rate of removal of aluminum is limited by the low

electrical conductivity of these media. Application of molten salt media, particularly those containing aluminum chloride, to the anodization of aluminum has been demonstrated in both alkali chloride - aluminum chloride (7,8) and organic salt - aluminum chloride (9,10) mixtures. Separation procedures using these types of materials provide a number of advantages over the use of other electrolytes. Foremost, the method is electrolytic in nature, and therefore provides a great deal of control in the attack. Secondly, the process results in formation of an additional amount of one of the components of the electrolyte, a chloroaluminate ion:



This process is not accompanied by gas evolution provided the electrode is not made so anodic as to decompose the melt. Finally, because the electrolyte used is a molten salt, the conductivity is high, allowing a rapid removal of material.

King, Brown, and Frayer (7) showed that the anodic dissolution of aluminum proceeds at 100% efficiency at 175°C in a 2:3 molar ratio NaCl-AlCl₃ melt over the current range 63-500 mA/cm². Recently, Gale and Osteryoung (8) found similar results in a 1:1 NaCl-AlCl₃ melt over the current range 0.03-6.30 mA/cm² at 175°C. Anodization of aluminum in an organic containing room temperature molten salt such as 2:1 AlCl₃-1-ethylpyridinium bromide was also reported to occur with 100% current efficiency (10).

This investigation was undertaken to examine the practicability of using these types of electrolytes to remove the bulk aluminum from a two phase Al-Al₃Ni matrix.

EXPERIMENTAL

All experiments were conducted in a glove box (Vacuum/Atmosphere Co., Model HE 43-6 DRI-LAB). The moisture content was maintained below 10 ppm; the oxygen concentration was determined to be between 10 and 25 ppm as measured with a calibrated light bulb (11).

1-ethyl pyridinium bromide (EPB) was prepared by refluxing ethyl bromide (Baker A.R. Grade) with 10% excess pyridine (Baker A.R. Grade) in acetone (Eastman) as the solvent for 5-6 hours. The resulting white hygroscopic crystals were suction dried, washed with anhydrous diethyl ether (Baker A.R. Grade) and vacuum dried at 34°C overnight. After drying, the salt was transferred to a stoppered storage bottle in the glove box. The melting point of the dried EPB was 121°C and compared favorably with the literature (12).

A 2:1 molar ratio room temperature melt of AlCl_3 -EPB was prepared by adding aluminum chloride (Fluka, A.G., iron free) to the EPB. Considerable evolution of heat accompanies the addition and precautions should be taken to keep the temperatures of the mixture below 50°C; otherwise, some EPB may decompose during the addition resulting in a mixture which is difficult to purify. All glassware containing this melt was painted black since experience in this laboratory and that of other workers (13) indicates that the material is photosensitive. A melt of 1:1 molar ratio AlCl_3 -NaCl was prepared by fusing the correct amounts of aluminum chloride and sodium chloride (Baker, A.R. Grade) at 175°C in the glove box.

Both of the melts were purified utilizing a constant current electrolysis procedure (14). After approximately 10 days of electrolysis both melts were clear and water-like. They exhibited no peaks with cyclic voltammetry at a glassy carbon electrode (Figure 1).

Constant current and controlled potential electrolysis were accomplished with a potentiostat/galvanostat (Princeton Applied Research Corp, Model 173). A voltage programmer (Princeton Applied Research Corp, Model 175) was coupled to the potentiostat/galvanostat for linear sweep voltammetry.

Samples of the Al-Al₃Ni matrix were obtained from RADC as 1.25 inch by 0.125 inch diameter rods. In addition, a pure sample of Al₃Ni alloy was furnished which had been produced by fusing stoichiometric quantities of aluminum and nickel. Electrodes were fashioned from the samples by wrapping aluminum wire around the ends of the specimens that were not immersed in the melt to provide electrical contact. The immersed area of the particular Al-Al₃Ni specimen under investigation was measured geometrically. The Al₃Ni electrode was quite porous, and only an approximate geometrical area, 1 cm², could be measured.

In a typical electrolysis experiment the Al-Al₃Ni matrix rod or a piece of Al₃Ni was used as a working electrode in a three electrode cell (Figure 2). The cell was constructed of Pyrex. Cell temperature for experiments with the 1:1 AlCl₃-NaCl melt was maintained to $\pm 0.5^\circ\text{C}$ by means of a platinum resistance controlled furnace. The counter electrode was a cup shaped piece of aluminum foil (Research Organic/Inorganic Co., m5n purity), while the reference electrode was a spiral aluminum wire (Alfa-Ventron, m5n purity) isolated from the melt by a 10 mm fine porosity Pyrex frit. All potentials described during this investigation were measured against this electrode, *i.e.*, the Al(III)/Al couple.

For experiments requiring a flowing electrolyte stream, a centrifugally induced convective stirrer (Figure 3) was constructed. When this

device was turned with an electrode rotator (Pine Instrument Co., ASR-2) at speeds above 50 RPM, a flowing electrolyte stream was present at the electrode suspended in the center of the stirrer.

RESULTS AND DISCUSSION

In order to determine the approximate potential range in which aluminum could be removed by electrolysis without appreciable damage to the trialuminum nickelide filaments, current-potential curves for the anodization of aluminum and trialuminum nickelide were constructed using linear sweep voltammetry. The working electrode was either pure aluminum wire or Al_3Ni , respectively. The potential of the working electrode was varied linearly from a potential of 0.0V vs. Al(III)/Al to the anodic limit of the melt under consideration. In the 1:1 $\text{AlCl}_3\text{-NaCl}$ melt at 175°C the practical limit is about 2.2V anodic of the aluminum reference electrode and represents the oxidation of chloride to chlorine (14). The useful limit of the 2:1 $\text{AlCl}_3\text{-EPB}$ melt is about 1.75V (Figure 1) and probably represents the oxidation of bromide to bromine. The potential at which attack on the Al_3Ni occurs in 2:1 $\text{AlCl}_3\text{-EPB}$ is about 1.0V (Figure 4) at the 20 mV/sec sweep rate used for these studies.

The voltammogram for aluminum anodization in the 2:1 $\text{AlCl}_3\text{-EPB}$ melt (Figure 4) exhibits an interesting feature. The shape of the current-voltage curve suggests formation of a passivating layer of aluminum chloride. Similar behavior has been obtained in other aluminum chloride rich melts (7,15). This feature was not observed for the 1:1 $\text{AlCl}_3\text{-NaCl}$ melt (Figure 5) during the time span of the voltammogram. Two factors may have contributed to the absence of passivation in the latter case. First, the melt is able to solvate approximately an additional mole of aluminum chloride per mole of electrolyte before forming a solid aluminum chloride layer. Second, thermal convection currents in the heated melt prevented formation of a sodium ion depleted diffusion layer. Therefore,

passivation behavior can be alleviated somewhat by using some type of forced convection. A special stirrer (Figure 3) was constructed in order to provide uniform forced convection. An experiment with this device is described below. Such a stirrer should provide other advantages as well; for example, a more complete stripping of aluminum from the interior of the exposed filament bundle as a result of the flowing electrolyte stream.

The relative amounts of material that can be removed anodically from an aluminum specimen in the two melts examined in this study can be judged by comparison of the linear sweep voltammograms at a given potential (Figures 4 and 5). At 0.8V vs. Al(III)/Al the current at an aluminum wire electrode in the 1:1 AlCl₃-NaCl melt was about 20 times that of a similar electrode in the 2:1 AlCl₃-EPB melt. Since the quantity of material that can be removed anodically is governed by Faraday's law, the weight in grams of aluminum, W, that can be removed by electrolysis in t seconds at a current of i amperes is:

$$W = 9.33 \times 10^{-5} it \quad [2]$$

For a typical specimen of 1.28 g containing 20 wt-% Al₃Ni as the rod phase, an electrolysis time of 15 hours in the 1:1 AlCl₃-NaCl melt and 336 hours in the 2:1 AlCl₃-EPB melt would be required to remove all bulk aluminum. These times were calculated for an unstirred solution assuming that the limiting current at the electrode is that reflected at 0.8V vs. Al(III)/Al from the linear sweep voltammograms. The values calculated represent an upper bound in time since stirring increases the current density. For example, Holleck and Giner (14) observed that the anodic current increased linearly with the square root of the rotation rate at a rotating aluminum electrode. Their study was an example of ideal forced

convection and such efficiency was not realized with the apparatus used in the present work.

Preliminary electrolysis experiments were accomplished in the 2:1 AlCl_3 -EPB melt by anodizing the Al- Al_3Ni matrix working electrode while stirring with a propeller. Portions of the rod which were not desired to be anodized were coated with heat shrinkable Teflon* tubing. A microphotograph of an unpolished Al- Al_3Ni matrix specimen prior to electrolysis (Figure 6) shows no evidence of exposed Al_3Ni whiskers. The same specimen was subjected to anodization by constant current at 0.16 A/cm^2 in a stirred solution of 2:1 AlCl_3 -EPB, removed and washed successively with dimethylsulfoxide, distilled water, and acetone. Microphotographs (Figure 7) revealed that bulk aluminum had been removed from the surface of the specimen and exposed Al_3Ni whiskers were present. Visual inspection revealed no discernible damage to the fibers. During electrolysis the potential of the Al- Al_3Ni electrode rose to $0.1\text{V vs. Al(III)/Al}$. An experiment at 0.54 A/cm^2 with 0.22V polarization produced the same result (Figure 8). At this current density the cell potential exceeded 3V vs. Al(III)/Al when stirring had been discontinued.

Al- Al_3Ni electrolysis was accomplished in the 1:1 AlCl_3 -NaCl melt in a manner similar to the procedure used for the 2:1 AlCl_3 -EPB melt. Electrolysis at a current density of 0.180 A/cm^2 in an unstirred solution resulted in a polarization of $0.3\text{V vs. the Al(III)/Al couple}$. Microphotographs revealed essentially the same result as that obtained previously, except that electrolysis was much faster and no stirring was required to offset passivation.

*Registered Trademark

The centrifugally induced stirrer (Figure 3) containing an Al-Al₃Ni rod was rotated at about 160 RPM during passage of a 0.10 A/cm² current. An apparent resistance polarization of 0.5V was noted indicating that the device had caused moderate shielding of the Al-Al₃Ni electrode within. Microphotographs (Figure 9) of a portion of the matrix sample after electrolysis showed that even removal of bulk aluminum from the specimen had taken place. At the outset of this experiment removal of the entire bulk aluminum phase was intended using this device. However, this could not be realized because the specimen rod was permeated with numerous voids (Figure 10) throughout the length of the specimen. These voids would not allow a fiber bundle to be formed for as the fibers were exposed they became detached from the rod and were deposited in the bottom of the cell.

CONCLUSION

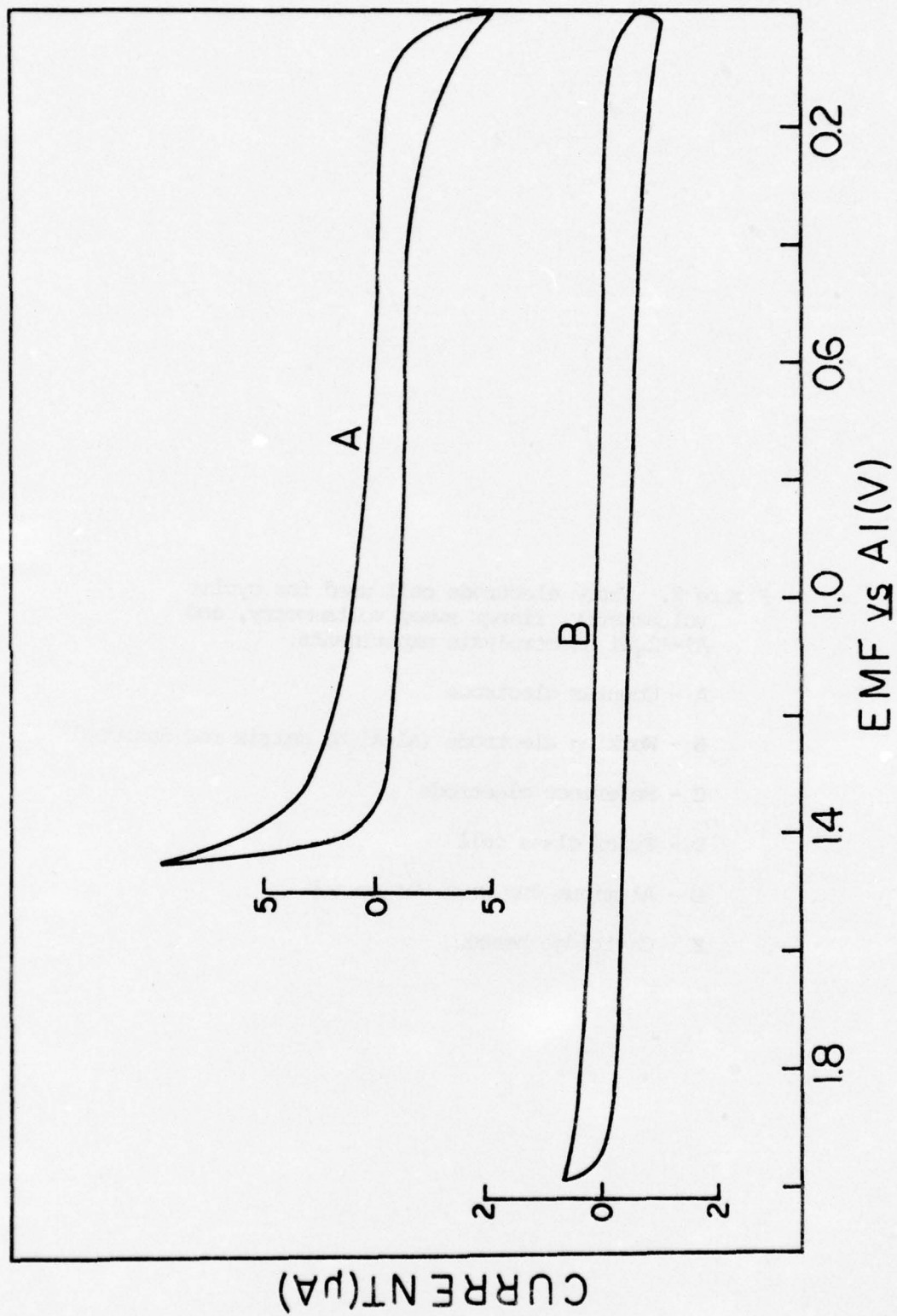
The electrolytic removal of aluminum from a two phase matrix of aluminum containing trialuminum nickelide whiskers has been demonstrated in two different molten salt electrolytes. Both electrolytes were shown to possess certain advantages and disadvantages. The 2:1 aluminum chloride-1-ethyl-pyridinium bromide melt was easiest to use since it could be prepared and maintained at room temperature. However, this melt suffered from severe passivation during anodization, *i.e.*, the high aluminum chloride content did not allow efficient solvation of additional aluminum chloride produced during anodization. In contrast, the inorganic melt, 1:1 aluminum chloride-sodium chloride, demonstrated no passivation in the current ranges examined in this study. This was attributed to the ability of 1 mole of 1:1 $\text{AlCl}_3\text{-NaCl}$ to solvate almost an additional mole of aluminum chloride. Unfortunately, this electrolyte was more difficult to use because it must be maintained at temperatures above its freezing point of 160°C .

A forced convection stirring device constructed for this investigation has been shown to be practical for complete removal of aluminum from a two phase specimen. However, this could not be demonstrated unambiguously because of imperfections present in the samples tested.

REFERENCES

1. B.J. Bayles, J.A. Ford, and M.J. Salkind, Trans. Met. Soc, AIME, 239, 844 (1967).
2. F.D. Lemkey, R.W. Hertzberg, and J.A. Ford, ibid., 233, 334 (1965).
3. Y.G. Nakagawa and G.C. Weatherly, Acta Met., 20, 346 (1972).
4. Dr. J. Hutta, Rome Air Development Center, Hanscom AFB, MA, personal communication (1975).
5. A.W. Davidson and F. Jink, J. Amer. Chem. Soc., 72, 1700 (1950).
6. W.E. Bennett, A.W. Davidson, and J. Kleinberg, ibid., 74, 732 (1952).
7. L.A. King, A.D. Brown, and F.H. Frayer, Proc. OAR Res. Appl. Conf., 21 March 1968, p. J-1.
8. R.J. Gale and R.A. Osteryoung, J. Electrochem. Soc., 121, 983 (1974).
9. F.H. Hurley and T.P. Wier, ibid., 98, 203 (1951).
10. W.H. Safranek, W.C. Schickner, and C.L. Faust, ibid., 99, 53 (1952).
11. Letter, 3 October 1968, General Motors Research Laboratories.
12. J. Willems and J. Nys, Bull. Soc. Chim. Belg., 66, 502 (1957).
13. L.L. Miller, Colorado State University, personal communication (1976).
14. L.G. Boxall, H.L. Jones, and R.A. Osteryoung, J. Electrochem. Soc., 121, 212 (1974).
15. G.L. Holleck and J. Giner, J. Electrochem. Soc., 199, 1161 (1972).

Figure 1. Cyclic voltammograms of the 2:1 AlCl_3 -EPB melt, A, and the 1:1 NaCl-AlCl_3 melt, B, at a glassy carbon electrode ($A = 0.215 \text{ cm}^2$), sweep rate = 100 mV/sec.



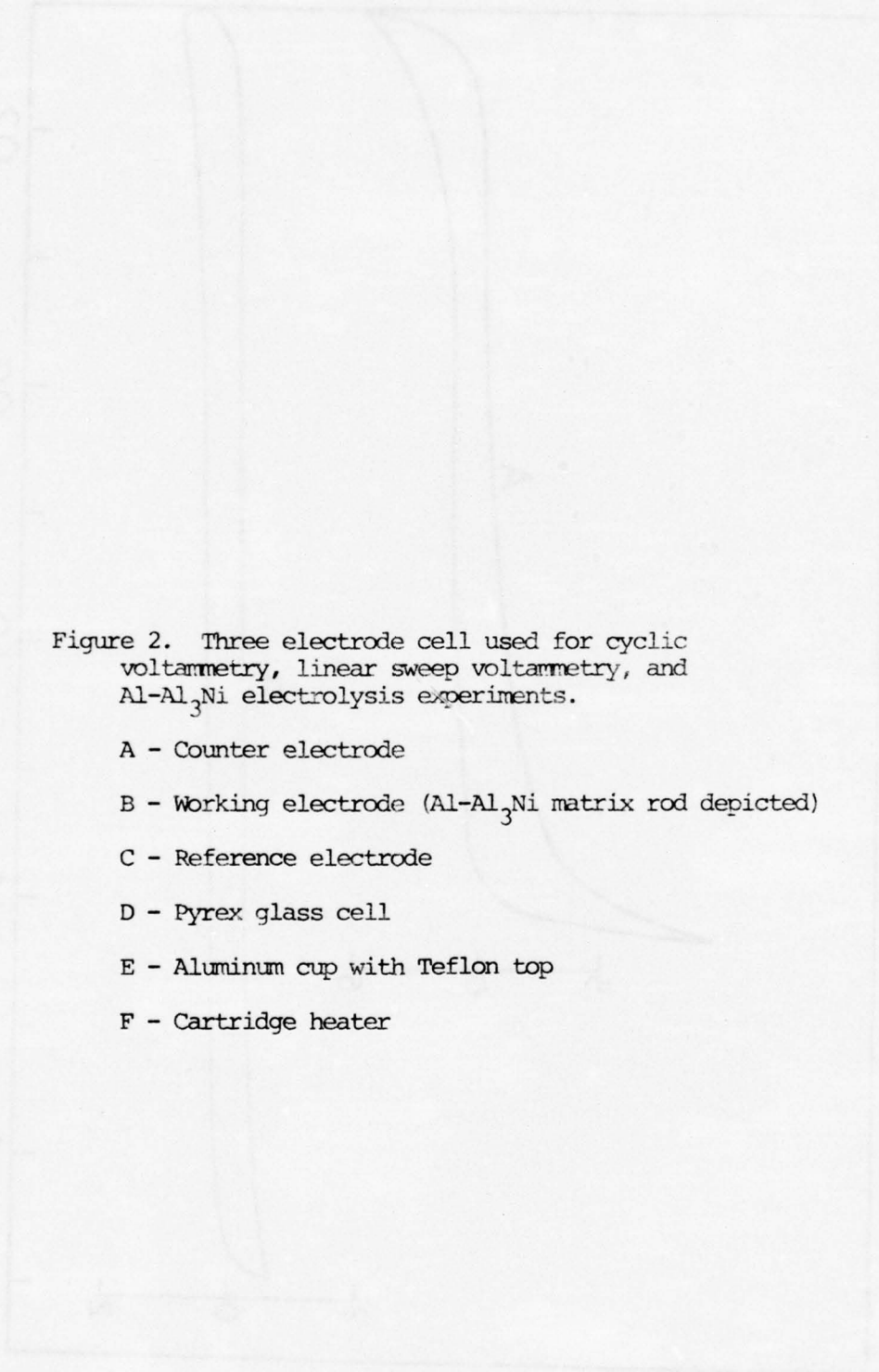


Figure 2. Three electrode cell used for cyclic voltammetry, linear sweep voltammetry, and Al-Al₃Ni electrolysis experiments.

- A - Counter electrode
- B - Working electrode (Al-Al₃Ni matrix rod depicted)
- C - Reference electrode
- D - Pyrex glass cell
- E - Aluminum cup with Teflon top
- F - Cartridge heater

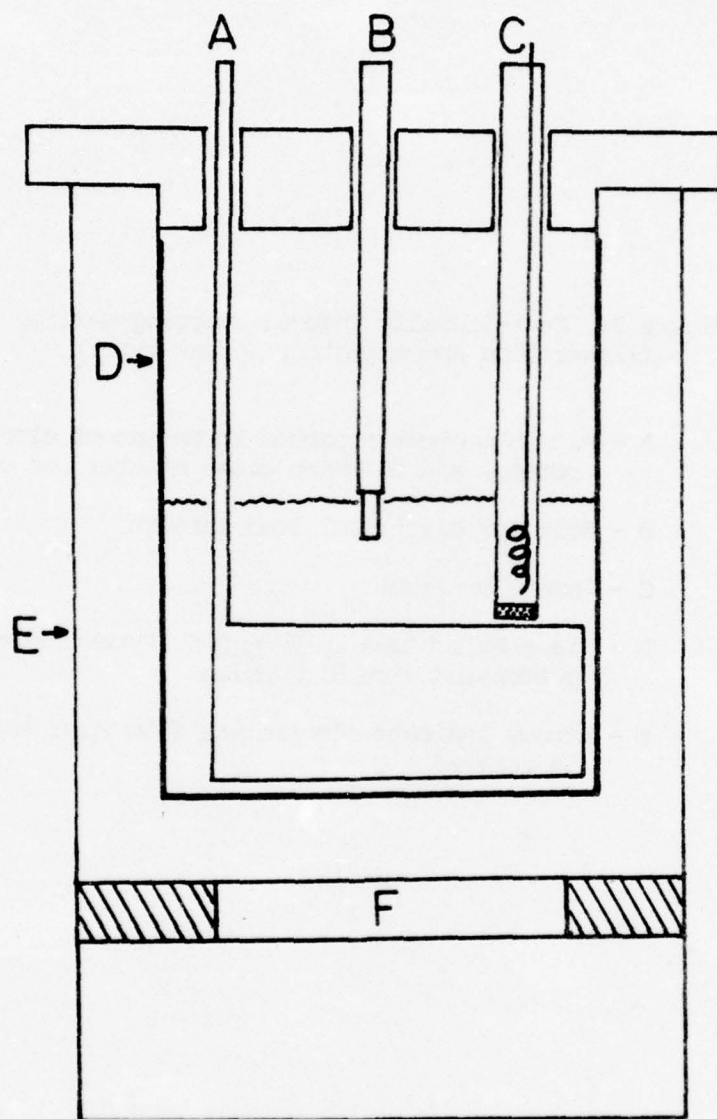


Figure 3. Centrifugally induced stirring device.
(Illustration approximately actual size.)

- A - Aluminum chuck attached to collet of electrode rotator and to which glass stirrer was cemented
- B - Hole for electrical lead through
- C - Immersion level
- D - Glass bulb blown in 7 mm O.D. Pyrex tubing and perforated with 3 mm holes
- E - Arrows indicate electrolyte flow when device is rotated

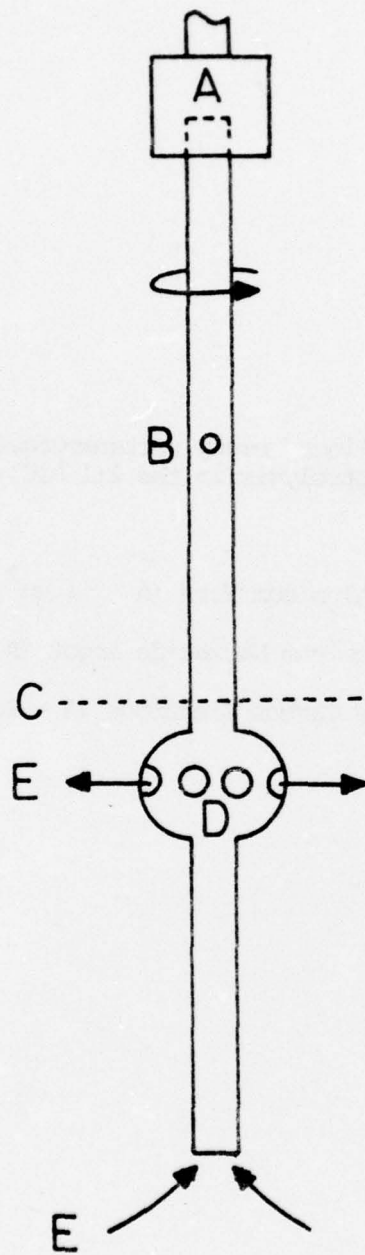


Figure 4. Anodic linear sweep voltammograms for Al and Al_3Ni electrolysis in the 2:1 AlCl_3 -EPB melt at 23°C

- Pure Aluminum Wire ($A = \sim 1 \text{ cm}^2$)
- Trialuminum Nickelide Ingot ($A = \sim 1 \text{ cm}^2$)
- Glassy Carbon Electrode ($A = 0.215 \text{ cm}^2$)

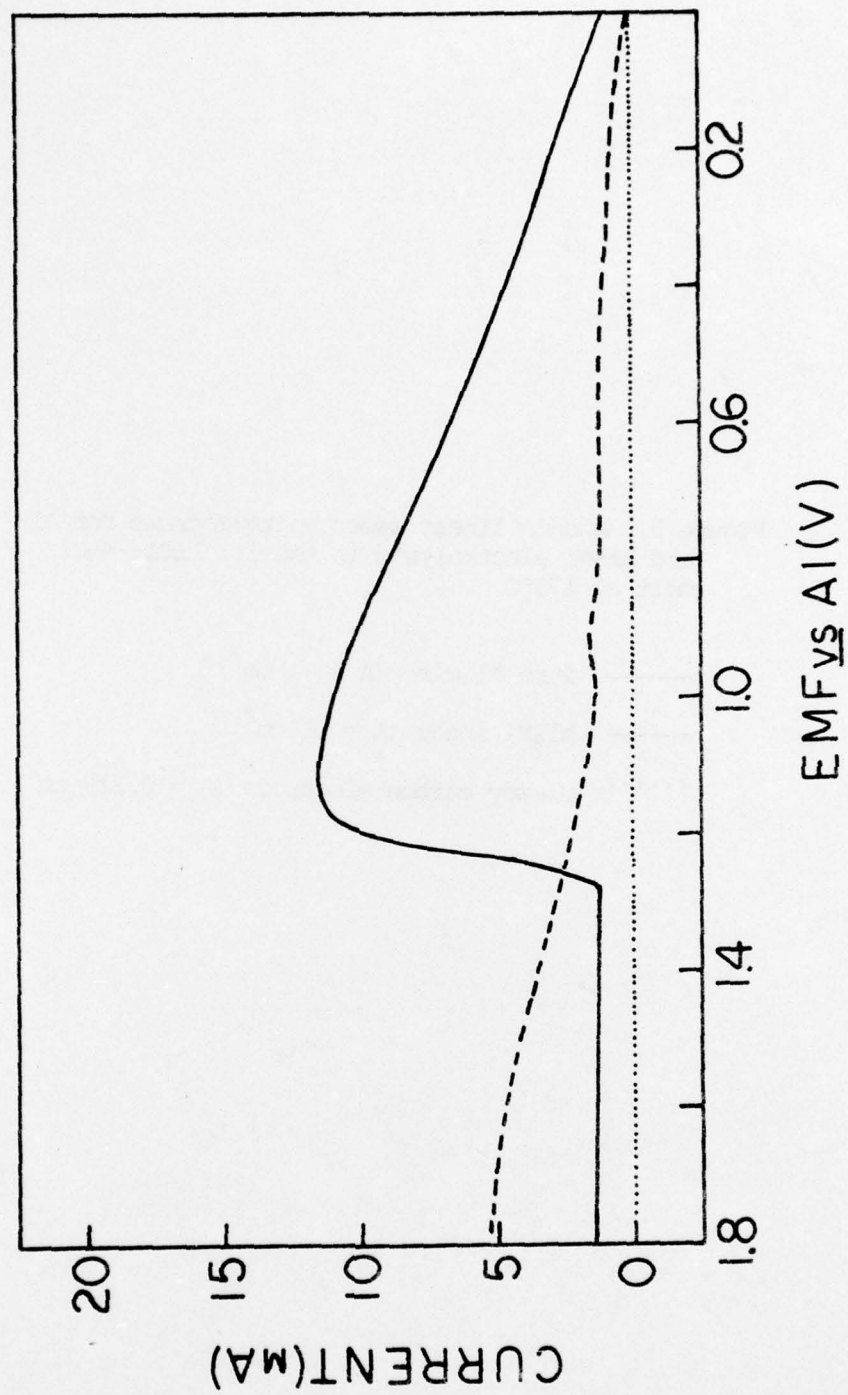


Figure 5. Anodic linear sweep voltammograms for Al and Al_3Ni electrolysis in the 1:1 $\text{AlCl}_3\text{-NaCl}$ melt at 175°C

- Pure Al wire ($A = \sim 1 \text{ cm}^2$)
- Al_3Ni ingot ($A = \sim 1 \text{ cm}^2$)
- Glassy carbon electrode ($A = 0.215 \text{ cm}^2$)

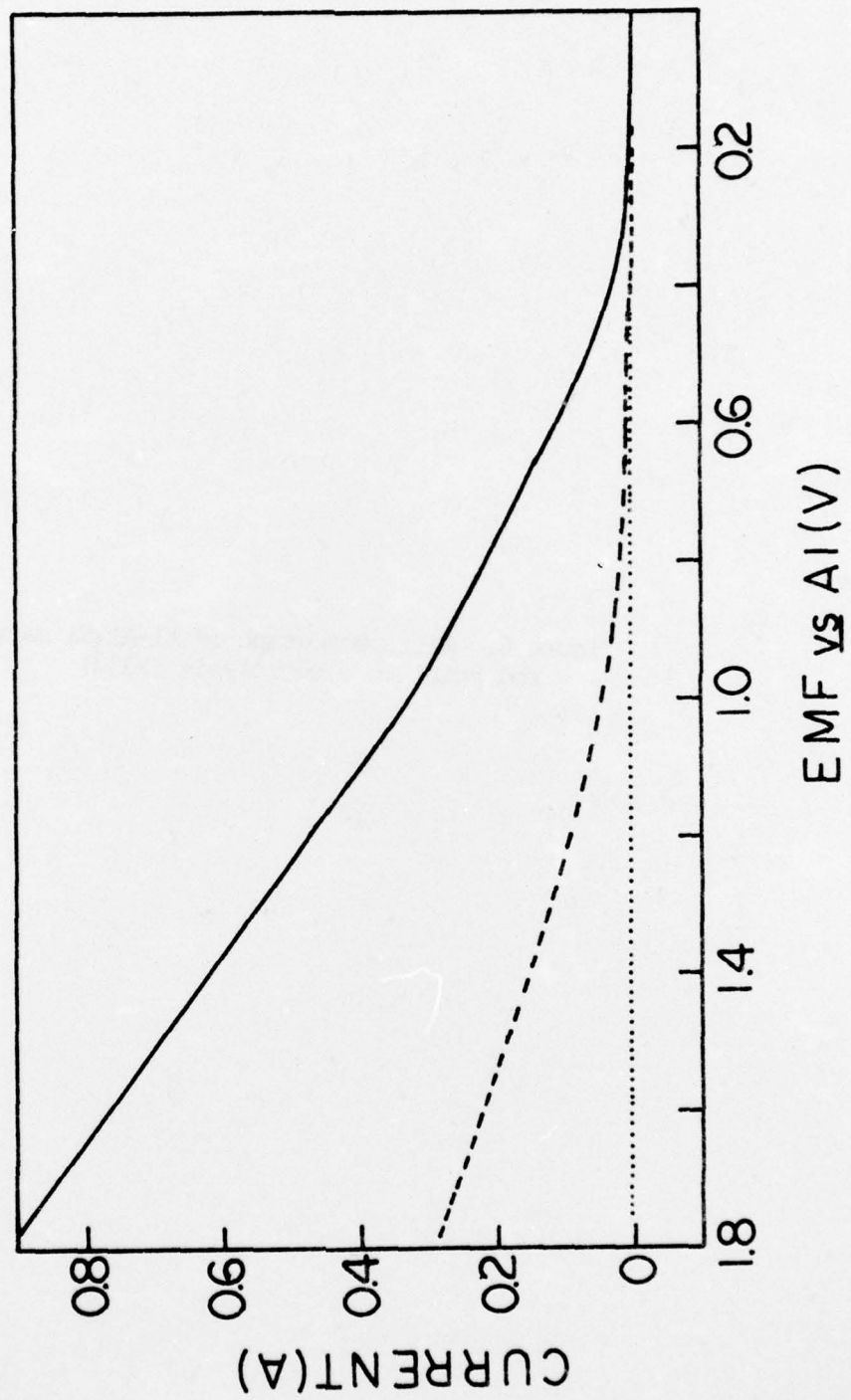


Figure 6. Microphotograph of Al-Al₃Ni matrix
rod prior to electrolysis (X350)

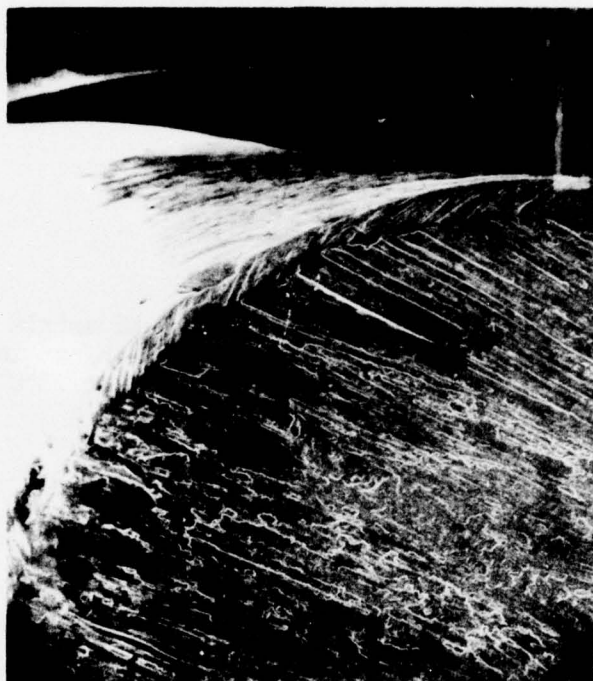


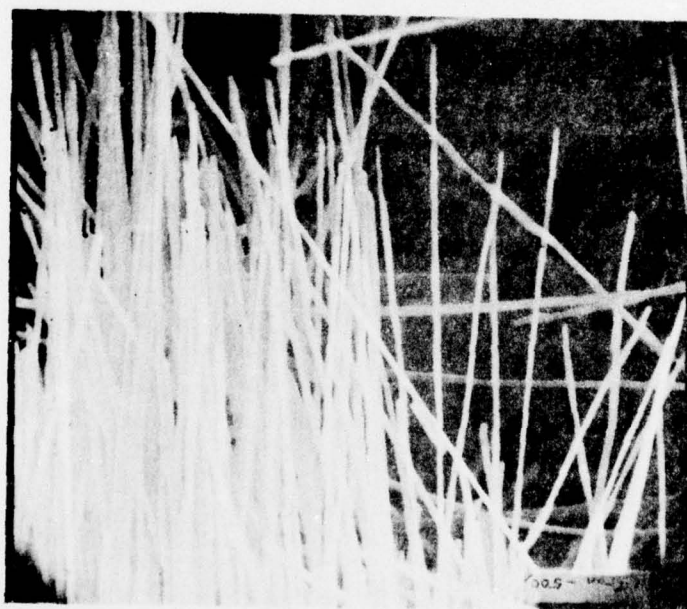
Figure 7. Microphotographs of Al-Al₃Ni matrix rod surface after anodization at 0.16 A/cm² in a stirred solution of 2:1 AlCl₃-EPB at 23°C

A X740

B X1860



A



B

Figure 8. Microphotograph of Al-Al₃Ni matrix rod surface after anodization at 0.54 A/cm² in a stirred solution of 2:1 AlCl₃-EPB at 23°C

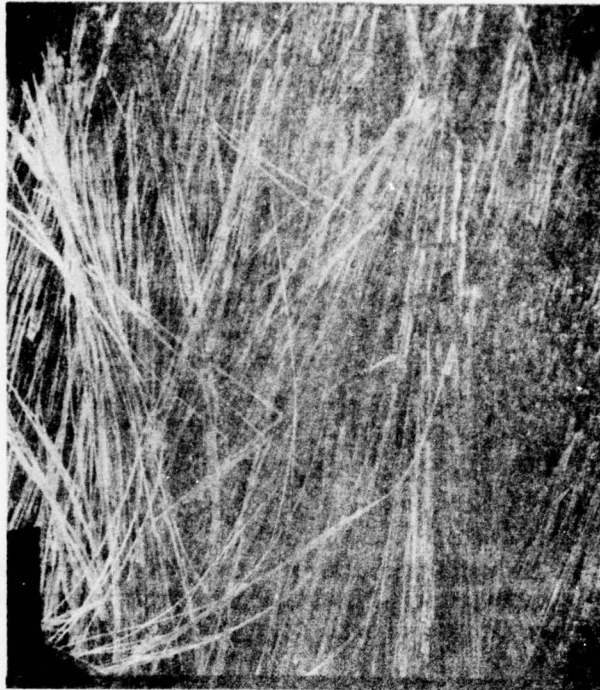
X2760



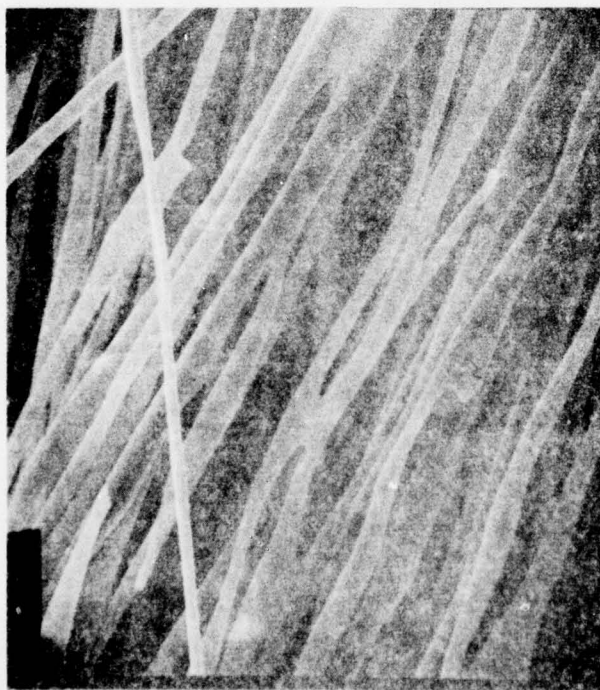
Figure 9. Microphotograph of Al-Al₃Ni matrix rod surface after anodization at 0.10 A/cm² with centrifugally induced stirring in 1:1 NaCl-AlCl₃ at 175°C

A X170

B X1360



A



B

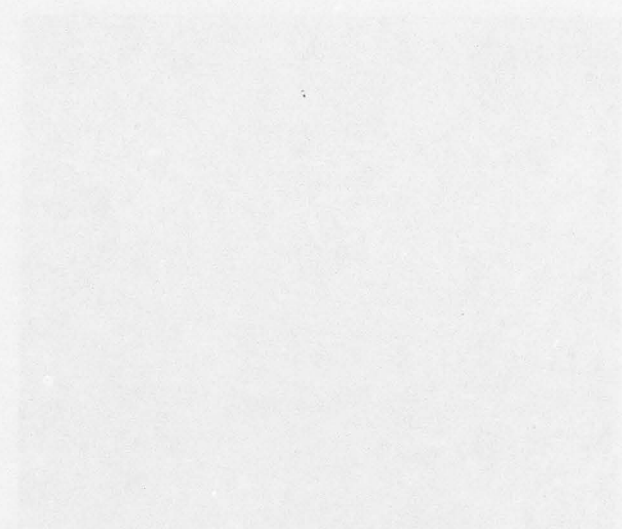


Figure 10. Microphotograph of Al-Al₃Ni rod surface after anodization illustrating void introduced into rod during growth of two phase matrix

X41

